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Jennifer R. Verkouteren
Greg Gillen
R. Michael Verkouteren
Robert A. Fletcher
Edgar S. Etz
George A. Klouda
Alim A. Fatah
Philip J. Mattson

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Jennifer R. Verkouteren

Greg Gillen

R. Michael Verkouteren

Robert A. Fletcher

Edgar S. Etz

George A. Klouda

*Surface and Microanalysis Science Division
Chemical Science and Technology Laboratory*

Alim A. Fatah

Philip J. Mattson

*Office of Law Enforcement Standards
Electronics and Electrical Engineering Laboratory*

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Carlos M. Gutierrez, Secretary

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Michelle O'Neill, Acting Under Secretary of Commerce for Technology
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U.S. Capitol Police
U.S. Secret Service

NOTE: All italicized words are defined, strictly for the purposes of this document, in Appendix 1. The definitions given in Appendix 1 are meant to facilitate the use and understanding of this document, which relates to IMS-based trace explosives detectors, and should not be taken as general definitions. Appendix 2 provides a glossary of explosives materials pertinent to this document, and is not meant to be selective and not comprehensive.

1. OBJECTIVES

The purpose of this document is to establish minimum performance requirements and an associated test method for Ion Mobility Spectrometry (IMS) based *trace explosives* detectors for use by the *first responder* community. Information concerning the theory and operation of IMS trace detectors is presented to enhance the understanding of the performance requirements, and to aid first responders in the selection of equipment that best suits their specific needs. The definition of first responder is considered broadly to include those who must work to prevent acts of domestic terrorism, in addition to those who must respond to emergency situations. An assessment of current and future needs for IMS trace detection was conducted through interactions with the people or organizations listed in the acknowledgements, and a summary is given in this document. Although some first responders already use IMS trace detection equipment, there are a wide variety of possible uses that may be contemplated for the future.

Although this document does not reference specific manufacturers or instrument models, much of the information has been derived from evaluations of the most current (as of the date of this document) models of IMS trace explosives detectors. The instrumentation available for evaluation includes both *desktop* and *handheld* IMS detectors, but does not include *portal* detectors. Pictures of these instruments and their associated materials, or data collected from the instruments, may be shown in various figures throughout the document, but the specific manufacturer or instrument model will not be identified.

A test method is provided in this document that addresses the basic performance criteria of desktop IMS trace detectors. The basic performance criteria are the minimum requirements that a detector must meet to qualify as an IMS trace explosives detector. The basic performance criteria may not be sufficient for all applications, and therefore a more complete set of criteria is also provided. It is incumbent on the user of the document to decide which of the additional criteria are necessary for the intended application of the equipment; some guidance is provided for generalized types of uses. A series of test results obtained by use of the method on the available instruments are presented in the document.

2. USES and NEEDS ASSESSMENT

The premise of IMS trace detection is that anyone handling explosives (or narcotics) will leave microscopic, invisible traces on anything they touch. These fingerprint residues will be present on personal items such as clothing and hair, and on objects such as luggage handles, laptop computers, door handles, steering wheels, etc. Such fingerprint residues commonly contain enough explosives (or narcotics) to be detected by IMS instruments as long as the material can be effectively sampled. Other applications may examine post-blast residues, but this is not the subject of this document.

Plastic bonded explosives such as C-4¹ and Semtex are mixtures of crystals of explosive with a sticky, plastic binder, as shown in Figure 1. A typical fingerprint residue produced after handling C-4 is shown in Figure 2, using polarized light microscopy to selectively highlight the RDX particles. The three particles of RDX marked by arrows in Figure 2 range in size from 50 to 90 μm in diameter; many smaller particles are present, some of which are barely visible in the image, and many others that are too small to be visible at this magnification. Collection of any one of the three marked RDX particles would provide more than enough material for a positive response by IMS, as most IMS trace detectors can reliably alarm on 3 ng or more of RDX (Figure 3), and a single 50 μm particle of RDX weighs approximately 100 ng (Table 1).

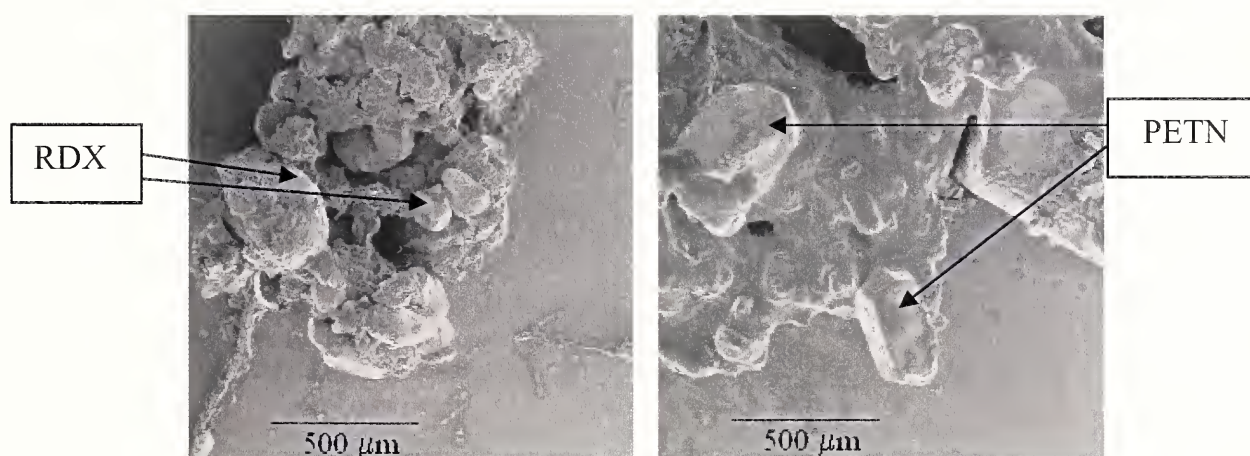


Figure 1. Scanning electron microscope images of C-4 (left) and Semtex (right) containing crystals of explosive (arrows) in a plastic binder.

¹ See Appendix 2: Glossary of Selected Explosives Materials.

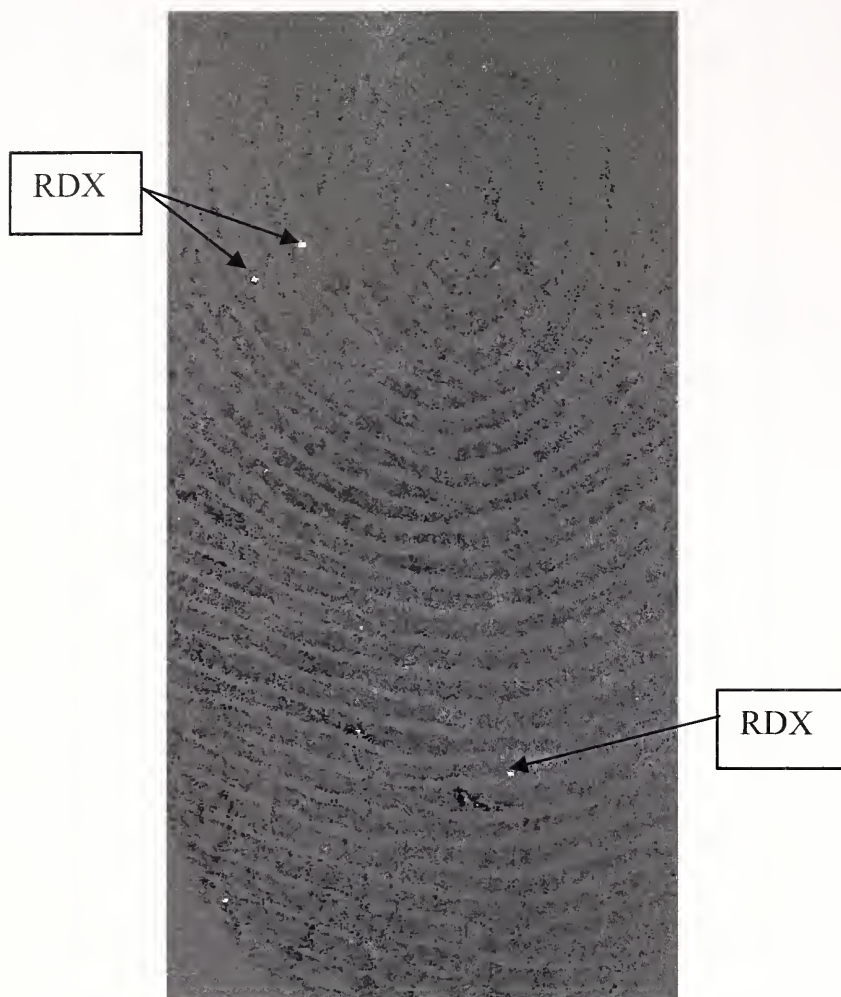


Figure 2. Fingerprint residue produced after handling C-4; RDX particles appear bright in the image, with the largest (50 μm to 90 μm) marked by arrows.

Table 1. Approximate masses of RDX and PETN particles with respect to particle diameter assuming spherical particles.

Particle diameter, μm	Particle mass
0.1	≈ 1 femtogram (0.000001 ng)
1	≈ 1 picogram (0.001 ng)
10	≈ 1 nanogram (1 ng)
50	≈ 100 nanograms (100 ng)
100	≈ 1 microgram (1000 ng)
1000	≈ 1 milligram (1,000,000 ng)

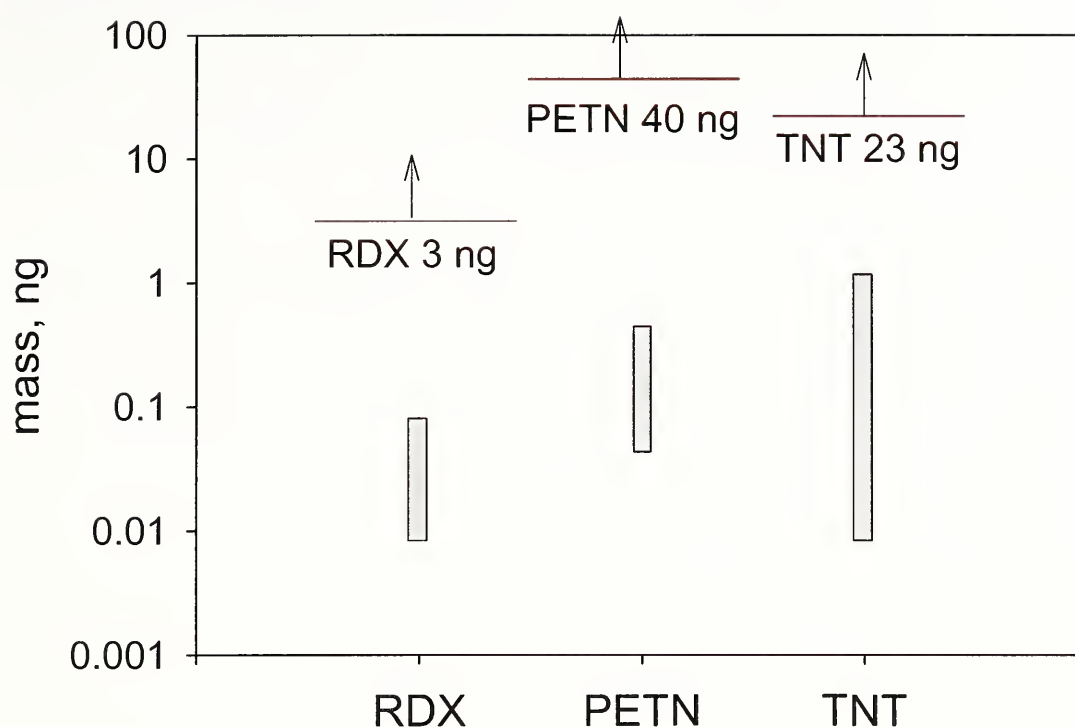


Figure 3. *Limits of detection* (boxes) obtained from commercial IMS instruments using solution standards of pure explosives (NIST Report of Analysis 837-36-04). Lines represent concentrations of explosives above which commercial IMS instruments should alarm 100 % of the time.

2.1 Examples of IMS usage

One of the most extensive uses of IMS trace explosives detection is for screening passengers and items at U.S. airports, conducted under the auspices of the Transportation Security Administration (TSA). The TSA has deployed thousands of IMS desktop instruments at security checkpoints, and has a general list of performance requirements. In particular, the TSA requires a high *throughput rate* because of the need to screen large numbers of people/samples, and so has a fairly low tolerance for *false positive alarms*. The U.S. military uses IMS trace explosives detectors to screen people, mail, vehicles, and other items at points of entry; their performance requirements include high *sensitivity* to the common explosives, particularly under field conditions and for realistic test samples. IMS detectors are used at entry points to government facilities to test vehicles and people for trace explosives. The U.S. Coast Guard uses IMS trace detection to screen cargo on ships, primarily for narcotics detection but also for explosives detection. Corrections facilities use IMS trace detection, again primarily for narcotics detection. Some state police agencies use IMS detectors to detect narcotics, explosives, and chemical warfare agents.

2.2 Generalized IMS usages

Some general categories of usage of IMS trace detection can be defined based on the information above. One category is a general screening usage, such as that employed by the TSA, where the

detector location is relatively fixed and people or objects are sampled as they pass by the screening station. Screening implies sampling a general population of people and/or objects for which the expectation of an actual occurrence of explosives is low. Screening applications are used for high-security events and at venues such as courthouses, sports arenas, public monuments, government facilities, etc. In general, the critical operational requirements include high throughput and continuous operation.

A second category of usage, like that employed by the U.S. Coast Guard, requires moving the detector to the area that must be sampled, such as onboard a ship. The requirements for a mobile usage of this type will probably include portability and ruggedness. IMS trace detection could be used for mobile applications in some of the ways that trained canines are used today. During routine traffic stops, a mobile IMS detector in the officer's vehicle could be used to detect explosives (or narcotics) on a driver's license or the suspect's car. Mobile IMS instruments could be used to check critical infrastructure such as bridges and tunnels by swiping likely surfaces during a sweep of the area. The domiciles or vehicles of suspected terrorists could be sampled to look for explosives and gain evidence for further action.

2.3 Advantages and limitations of IMS

IMS instruments are widely used for trace explosives detection, and compared with other types of analytical instruments are fast, relatively inexpensive, and can be used in a wide variety of environments with relative ease by people with minimal training. IMS instruments are generally easy to install, can be relatively mobile, and can have very high duty cycles. IMS instruments are widely deployed by various federal agencies, and the Transportation Security Administration (TSA), in particular, is working with the manufacturers towards improving the accuracy and reliability of the instruments.

The primary limitation of IMS for trace detection of explosives is that vapor collection is generally ineffective due to the low volatility of many explosives, and although operating in a sniffer mode would be best in many situations, it is not realistic. IMS instruments for explosives detection are best used in particle sampling mode. In addition, IMS is not as definitive for identification of explosive compounds when compared with other types of analytical instruments, and there are innocuous materials that can be falsely identified as explosives. Although IMS instruments for trace detection are relatively inexpensive, there are consumable costs that may become considerable if the units are operated continuously and/or in humid environments. Rigorous adherence to the recommended maintenance procedures is necessary to maintain performance, and our experience shows this may involve considerable attention and time, although not more than is expected in general for analytical instruments.

IMS instruments are capable of detecting a wide variety of compounds, which is one of the strengths of the technique, but the instruments cannot be simultaneously optimized for all compounds of interest. The IMS instruments reviewed for this document are optimized primarily for RDX, in that the concentrations at which instruments will alarm for this explosive are lower than for PETN or TNT (Figure 3) [data from NIST Report of Analysis 837-36-04]. As generally configured, IMS is not suitable for detection of many gunpowders, particularly black powder,

although some smokeless powders may be detected by components other than the primary propellant.

3. THEORY OF OPERATION

3.1 Background information

Instruments utilizing Ion Mobility Spectrometry (IMS) detect and identify samples as gases, either by directly sampling gases from the air or by heating solid particles to form vapors. [A schematic of an IMS instrument is given in Figure 4.] The gases are normally ionized with a radioactive material, usually nickel-63 (^{63}Ni), safely encased in the detector. The *selectivity* of the ionization process (i.e. the preferential ionization of the vapors of interest) is improved by means of an additive (a reactive ion dopant) present in the dry air stream circulated through the instrument. The selectivity can also be improved by the addition of a semi-permeable membrane that allows transit of only the vapors of interest into the ionization region. The *analyte* gas molecules form cluster ions that are allowed to drift through an electric field at atmospheric pressure between an electronic gating shutter and a collector against a countervailing flow of gas. Drift times are commonly less than 15 milliseconds and are a function of the mobility of the cluster ions, hence the name ion mobility spectrometry. The data are evaluated as *ion mobility spectra*, also known as *plasmagrams*, which are plots of peak intensity as a function of drift time. The positions of the peaks of many chemical compounds, including explosives, narcotics, and chemical warfare agents, have been characterized for the commercial IMS instruments.

The IMS process described above is known as linear IMS, since the cluster ions travel straight to the collector. Another recent development is differential IMS, where the ions travel in a zig-zag fashion by application of an intermittent orthogonal electric field through the drift region, which separates the ions based on their mobility differences during the electric field cycles. This method does not use a gating shutter and may have improved sensitivity over linear IMS.

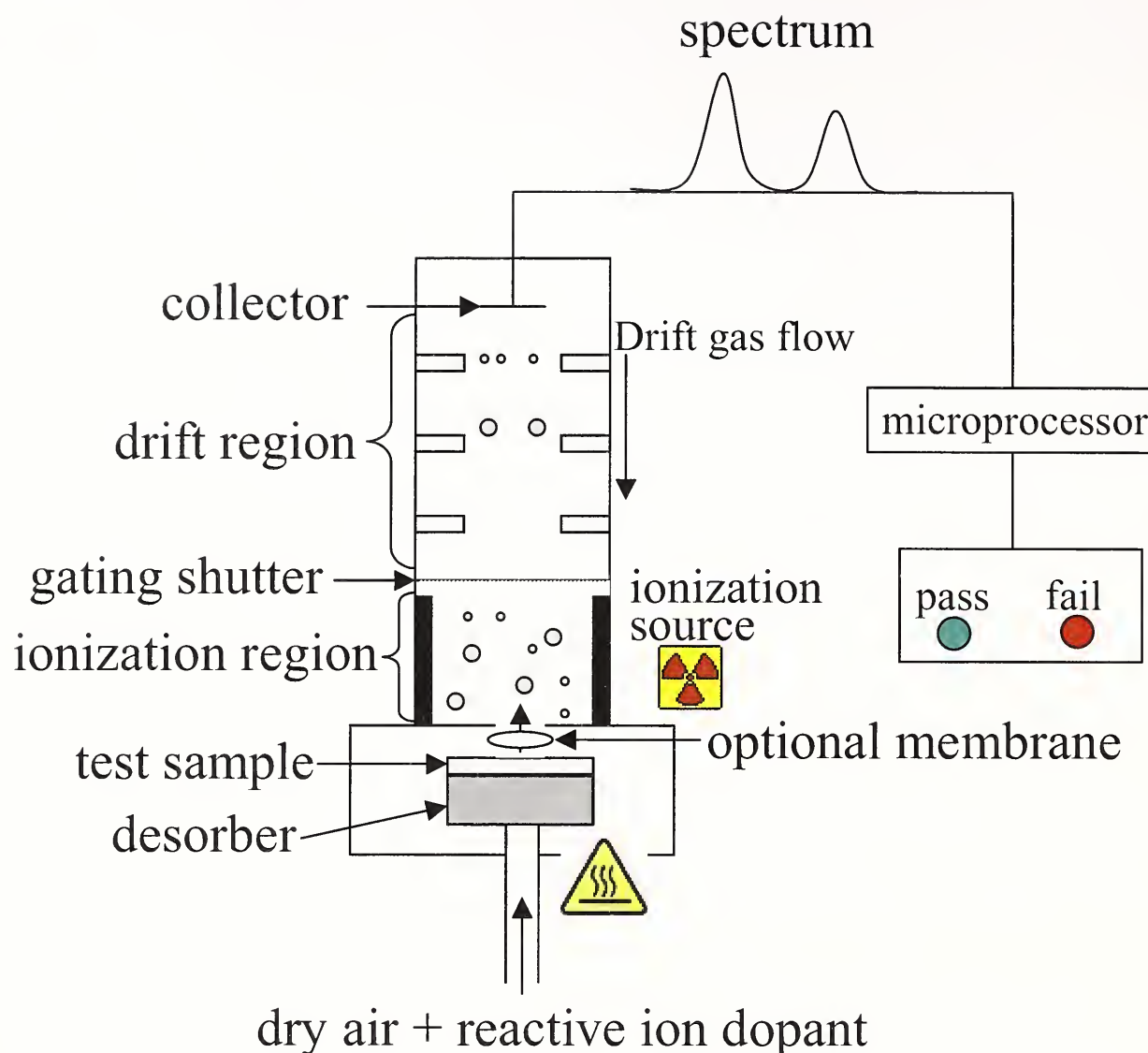


Figure 4. Schematic representation of an IMS instrument. Ionized gas molecules shown by grey circles.



Figure 5. Sample swipe method of particle collection.

3.2 IMS instrument categories

IMS detectors fall into three general categories based on physical configuration: 1) walk-through portals, 2) desktop units, and 3) handheld units. In addition to differences in size, these three different configurations have their own sample collection procedures. Walk-through portals are designed to dislodge particles of explosives from the body or clothing as people pass through. Desktop units require a cloth or paper of some type that is swiped over a surface to collect any particles of explosive (Figure 5). Handheld units are designed to collect vapors directly from the air, but may also accommodate a desktop-like swipe collection. At the current time, desktop units are the most

prevalent category of IMS instruments used as trace explosives detectors. Portal units are still primarily in the evaluation stage, and handheld units that rely on direct vapor sampling are limited in the number of different explosives that can be detected.

For explosives detection, IMS is much better suited to the analysis of particles rather than direct vapor analysis, because many high explosives produce relatively little vapor under normal environmental conditions. The *vapor concentrations* produced by solid samples of explosives over the normal range of ambient temperatures are quite low for RDX, PETN, and HMX when compared with EGDN, NG, and DNT (Figure 6) because of differences in *vapor pressure*. As a consequence, it is difficult to detect RDX, PETN, and HMX by direct vapor analysis. To resolve this problem, particles are collected for the portal and desktop units and heated with an internal heater, called a *desorber*, to produce the gases necessary for IMS detection. The optimal desorber temperature varies for each explosive type, but for practical purposes each manufacturer sets the desorber temperature to a fixed value (somewhere between 180°C and 230°C). A different (higher) desorber temperature is typically used for narcotics detection.

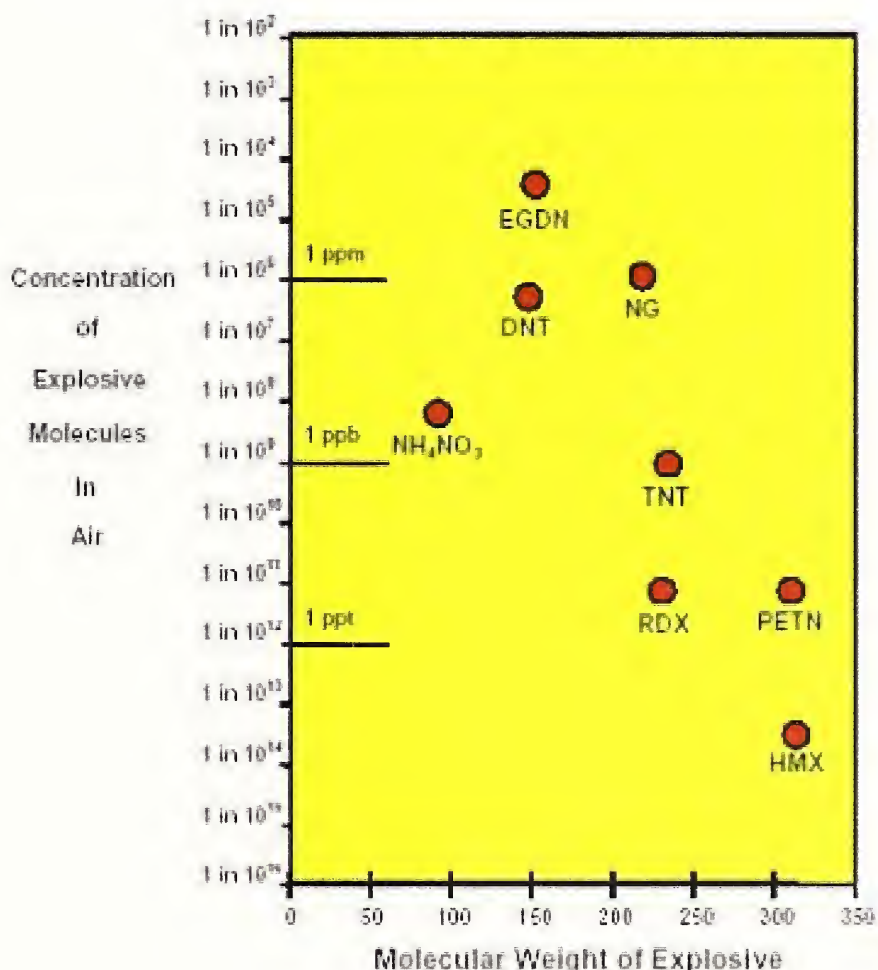


Figure 6. Vapor concentrations of pure high explosives in saturated air at 25 °C. Figure reproduced by permission from NIJ Guide 100-99.

3.3 IMS identification of explosives

All IMS instruments must have a library (spectral database) of peaks that serve to identify the explosives that the instrument is capable of analyzing. A chemical compound will generally produce multiple peaks, representing different molecular clusters with the reactant ions, and typically the IMS software uses the peak with the largest intensity for identification. Some IMS instruments use multiple peaks for compound identification. For example, the ion mobility spectrum shown in Figure 7 contains two peaks for RDX; the instrument that produced this spectrum requires both peaks for positive identification of RDX. The instrument manufacturer establishes a window that brackets an explosives peak, and when a sample is analyzed, the intensities in that window in the unknown spectrum are interrogated to determine whether a peak is present. IMS instruments will generate a *positive alarm* for peaks that are intense enough to exceed some specified *threshold level*.

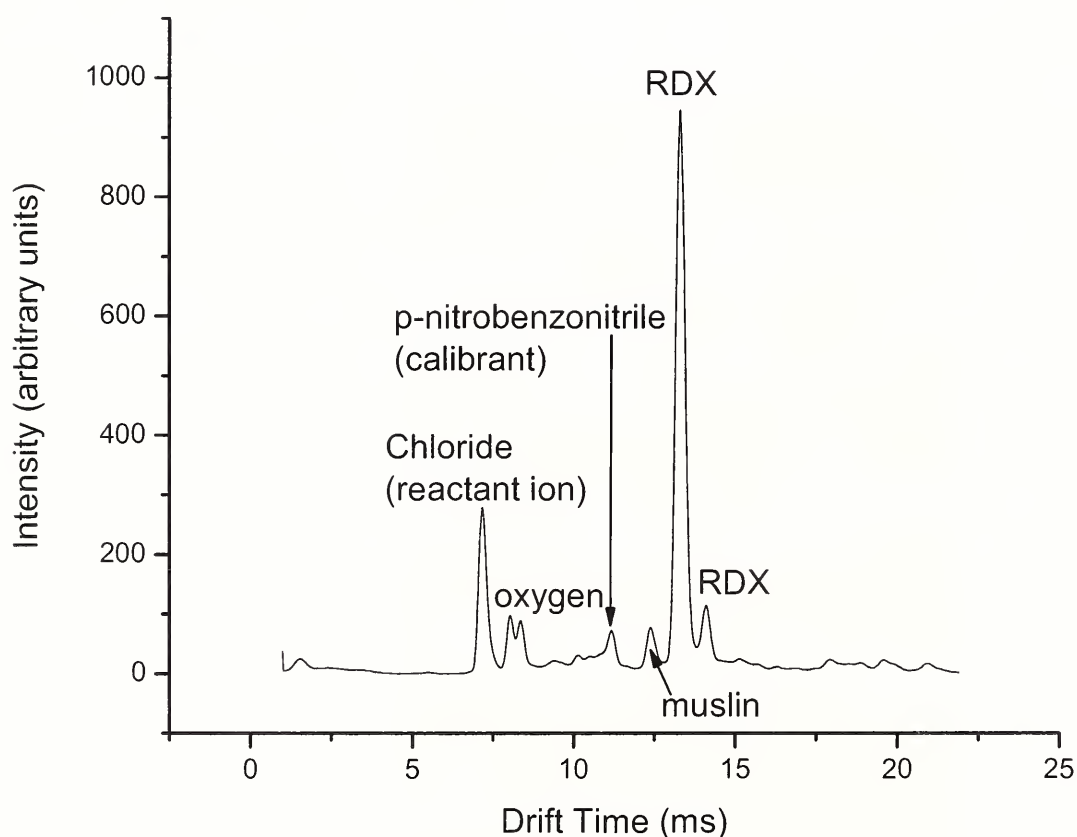


Figure 7. Ion mobility spectrum of RDX on muslin cloth.

Explosives are detected either by direct detection of the explosive, as for RDX, PETN, and TNT, by detection of a *marking agent* purposefully added during manufacture, such as EGDN, or by a component of the explosives formulation. Multi-base smokeless powders, one of the categories of gunpowder propellants, may be detected by the nitroglycerin (NG) component, or by DNT, which is added as a flash inhibitor. Ammonium nitrate (AN) is an oxidizer that may form part of an explosive mixture; only the nitrate portion of the compound is typically detected. Some IMS instruments will alarm for specific plastic-bonded explosives, such as C-4 and Semtex.

3.4 IMS calibration

The drift time of an ion, and hence its peak position, is affected by environmental factors, including the temperature, pressure, and humidity of the air in the drift region. Drift region temperatures are controlled by heaters that are normally set to a fixed temperature by the instrument manufacturers. Atmospheric pressure, on the other hand, cannot be controlled and will vary with elevation and with changes in weather. For this reason, a *calibration* procedure is needed to compensate for changes in peak position by correcting drift times with an internal *calibrant*. All IMS instruments must have some method of restricting water vapor from entering the ionization region of the detector. This is usually accomplished by using a *desiccant*, which must be monitored and replaced periodically. Some IMS detectors are designed with a membrane that is impermeable to water at the sampling inlet; these detectors still require a desiccant, although replacement is less frequent. Membranes tend to remove materials that might interfere with the analysis and therefore improve *selectivity*, but at the expense of *sensitivity*. Most IMS detectors can be operated in the normal ranges of relative humidity, but may require frequent changes of desiccant under high relative humidity conditions.

An ion mobility spectrum always contains peaks in addition to those from the target analyte that arise from the reactant ion and carrier gas, any internal calibrants, and, in the case of desktop detectors, from the sample swipe. The ion mobility spectrum of RDX shown in Figure 7 has many of these additional peaks identified; in this example a muslin fabric sample swipe has been used. The extent to which any additional peaks overlap with the analyte peaks will affect the ability of the IMS instrument to accurately determine the presence, or absence, of one or more explosives, and the type of explosive.

The quantity of explosive that will generate a positive alarm will depend on a number of factors, and will vary for the different explosives. Each instrument will have a recommended threshold for each explosive, defined in terms of peak intensity, above which a positive alarm will be produced. Well below the threshold level, the lowest amount of each explosive that can be reliably detected is known as the *detection limit*. The detection limit is typically determined under ideal laboratory conditions and is consequently much lower than the minimum amount that can be detected on a person or a surface.

IMS detectors can be operated in either the negative ion or the positive ion mode, as both types of ion are formed in the ionization process. Explosives are typically analyzed in the negative ion mode, and narcotics are typically analyzed in the positive ion mode. Those IMS instruments that provide for a *mode switch* from explosives to narcotics detection must allow for this switch in detector polarity and provide appropriate calibrants for both classes of materials. A higher desorber temperature is typically used for narcotics, as compared with explosives, and some time may be required for heating or cooling following a mode switch.

3.5 Sampling issues

Sample collection is perhaps the biggest challenge in detecting explosives in real-life situations, and knowledge of the physical behaviors of explosive compounds and likely areas for sampling are paramount for effective screening. The manufacturers of IMS instruments have developed several approaches for sampling either particles or vapors that cater to different needs and applications. The sample swipes for desktop IMS instruments can vary in both material and shape, with wands or other devices for holding the swipes. The vapor collectors and portals also employ a variety of approaches for sample collection.

When sampling for particle residues using swipes, it is recommended to rub hard on the surface and to attempt to get any residue onto the center of the swipe, or that portion of the swipe that will be in the center of the desorbed area. The manufacturer should indicate where the target area is on the swipe for maximum detection. The TSA is working to develop wands for holding the swipe that will inform the user when sufficient pressure has been applied. In addition, there is active research in determining those articles or areas that represent the best choice for sampling, either because they tend to retain residues best or because they are more likely to have come in contact with explosives.

3.6 Practical issues

All IMS instruments should be operable by trained, non-technical personnel, but note that the user interface, operating controls, and data recording procedures vary in complexity for the different instruments. Some manufacturers provide more access to default settings, such as threshold levels, desorber temperatures, etc. While it is useful for the knowledgeable operator to have access to these settings, particularly during calibration procedures, or to investigate errors such as false positive alarms, it is also necessary to be able to block access to these settings to maintain a consistent operating procedure during routine use.

There are a number of consumable items that are required for all IMS instruments, including sample swipes, desiccant, internal calibrants, dopants, gloves, membranes, batteries, etc, and these consumable costs will vary among the detector systems. Some IMS instruments can analyze explosives and narcotics simultaneously, whereas others require switching detection modes and repeating the sample collection; switching detection modes may require a considerable time for restabilization. IMS instruments operate from a standard AC power supply, and many also operate from batteries. The operating time on battery power will vary.

3.7 Potential problems

Chemical compounds that have the same drift time as one of the known explosives can trigger false positive alarms. Such interfering compounds are known to exist and can be present in relatively common products such as some perfumes and hand creams. In addition, the wide range of possible sample swipes, most of which have not been thoroughly characterized, present a possible source of false positives. *False negatives* can result from the presence of compounds that are preferentially ionized and may, therefore, prevent ionization of the explosives; these compounds are called masking agents. One way to diagnose such a problem is to observe the ion

mobility spectrum to determine whether a peak for the reactant ion is present. The absence of a reactant ion peak in the spectrum can signal the presence of a masking agent.

Dirt, oils, and other environmental contaminants may be sampled with the explosives, complicating the analysis and leading to false positives or negatives. In particular, oils are known to interfere with the analysis of narcotics by IMS. False positives and false negatives can occur from a change in the drift time of a compound, which can result from clustering (grouping) of the analyte gas molecules with water and other molecules, or from the presence of additional molecules in the drift region. In addition, simply changing the preset threshold levels, which are set somewhat arbitrarily with respect to detection limits, can alter the frequencies of false positives and false negatives.

Nuisance alarms arise from the presence of materials that produce a true positive alarm, but where the presence of those materials does not represent a threat. Both ammonium nitrate and nitroglycerin are used in bomb making and will alarm on most IMS instruments, but they have innocuous, legitimate uses as fertilizer and heart medicine, respectively. Nuisance alarms do not necessarily indicate a fault with the instrument, as the determination of the intended use of the material is beyond the scope of any analytical instrument designed strictly for compound identification. Nuisance alarms can also arise from contamination due to inadequate housekeeping or other sample handling issues.

4. GENERALIZED OPERATIONAL PROCEDURES

There are operational procedures common to all IMS trace instruments that serve to establish the throughput rate. A schematic is given in Figure 8 that describes an idealized procedure for a functioning IMS instrument. The terminology used in Figure 5 has been set for the purposes of this document to provide a common platform from which to describe IMS operations. Although the concepts are common to all instruments, the language used by the different manufacturers may vary from that used in Figure 8.

From a cold start, the first step that typically occurs is to heat any internal heating surfaces, such as the desorber, to establish thermal readiness (Step A-B in Figure 8). After thermal readiness is established, an *auto-calibration* is conducted (Step B-C) and the instrument indicates either a ready status or a fault, depending on the result of the auto-calibration. After a successful autocalibration, a *manual calibration* is typically performed (Step C-D) to verify the positive alarm, and if successful, a blank sample is analyzed to verify a return to background levels (Step D-E). At this point the detector is in a state of operational readiness and is ready to accept samples.

The time that it takes to move from Step A, the cold start, to Step E, operational readiness, is called the *startup time*. The *operational cycle* is governed by sampling and analysis (Step E-F), and *return to readiness* (Step F-E). In the absence of any alarms, the throughput rate is determined by the operational cycle time. Following any positive alarm, the background level must be reestablished to return to readiness. If a large amount of explosive has been introduced into the instrument, it may take considerable cleaning of the exposed surfaces, or a bake-out, to return to readiness. Whenever a mode switch is desired, such as to switch from explosive-to-narcotic sampling (if this is a feature of the detector), the operational cycle is terminated and usually the system must reestablish thermal equilibrium (Step G-B). The steps involved in establishing operational readiness (Steps B-E) must be repeated, typically with a different external calibrant more suitable for the new mode.

If at any point the system does not progress successfully to the next stage, the ideal procedure shown in Figure 8 is aborted and the system fault must be investigated. In addition, there are normal maintenance requirements for IMS trace detectors that include routine monitoring and replacement or refurbishment of desiccant, and routine cleaning of outer surfaces to prevent contamination. Some detectors will also require cleaning and/or replacement of membranes.

Time Schematic of Explosives Analysis

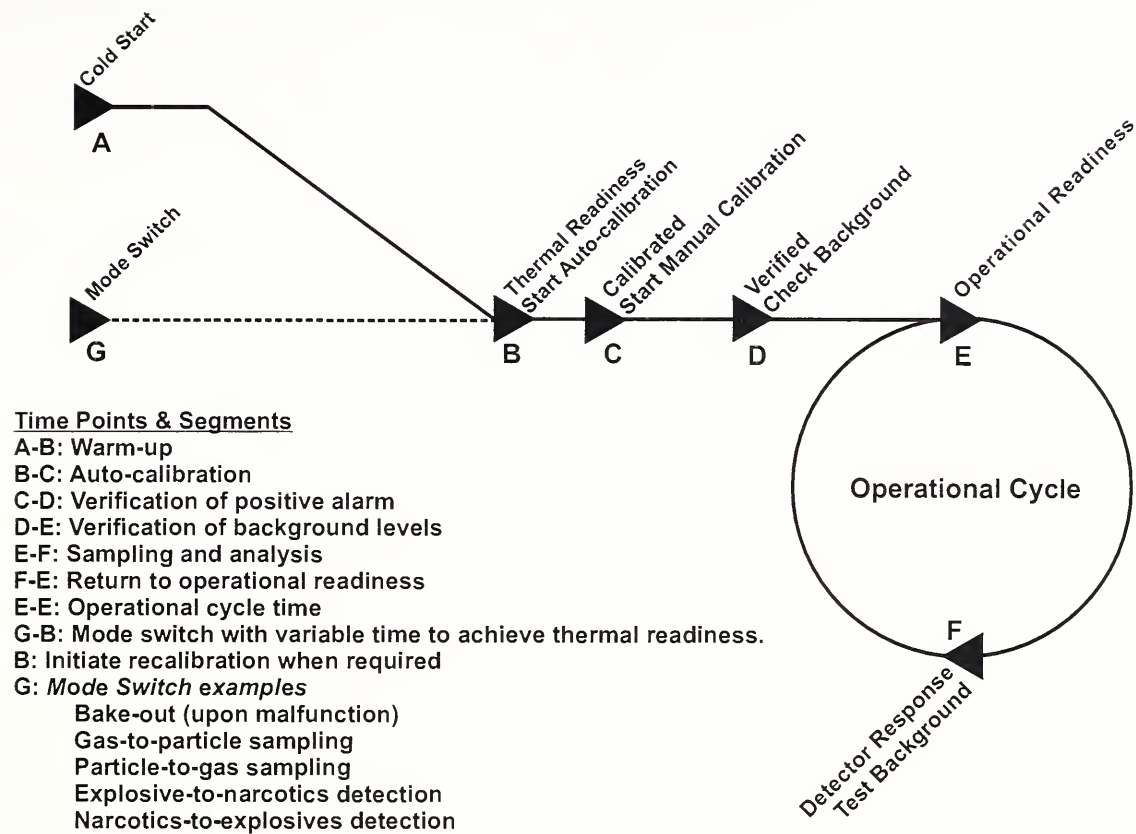


Figure 8. Schematic for the time sequence of explosives analysis by IMS.

5. OUTLINE OF INSTRUMENT SELECTION ISSUES

A summary of the primary issues for selection and testing of an IMS trace explosives detector is given below. Detailed information about these selection issues is given in Sections 3 and 4 of this document, and acceptance criteria for IMS trace detectors are given in Section 6 of this report.

1) **Determine the type of material to be identified:**

High explosives (HEs) : RDX, PETN, TNT and the plastic-bonded explosives C-4 and Semtex formulations

Use basic acceptance criteria in section 6.1.

Low explosives: gunpowder (smokeless), High explosives: AN, EGDN, HMX, NG, TATP, Tetryl

Narcotics: Cocaine, Heroin, Amphetamine, etc.

Also use additional acceptance criteria in sections 6.2.1 – 6.2.3

2) **Determine category of IMS detector/mode of collection:**

Desktop detector/particle collection

Use basic acceptance criteria in section 6.1

Handheld detector/particle collection (mobile application)

Also use additional acceptance criteria in sections 6.2.6 - 6.2.8

Non-covered application

Vapor collection

Portal detector

3) **Identify additional needs**

Screening application

Use basic criteria in section 6.1 and additional criteria in sections 6.2.9 – 6.2.15

Higher accuracy

Use basic criteria in section 6.1 and additional criteria in sections 6.2.4 – 6.2.5

3) **Test IMS detector**

For basic performance criteria (particle collection and detection of RDX, PETN, and TNT)

Use test method in section 7

No test method is provided for any additional uses.

6. REQUIREMENTS FOR ACCEPTANCE

6.1 Basic Performance Criteria

6.1.1 Compounds Detected

The instrument must be able to detect the following high explosives (HEs): RDX, PETN, and TNT

6.1.2 Minimum Amount Detected (Sensitivity)

The system must be able to produce a positive alarm in response to each of the following HEs at the stated minimum concentrations; 3 ng RDX, 23 ng TNT, and 40 ng PETN².

6.1.3 Return to Readiness

After a positive alarm, the system returns to background levels, as demonstrated by the absence of a positive alarm when a sample blank is analyzed.

6.1.4 System Calibration

The system must have a method of auto-calibration that compensates for environmental parameters such as barometric pressure.

6.1.5 Alarm

The system has either an audio or a visual alarm, or both, to indicate that an explosive has been detected, and tells the user what type of explosive has been detected. An ion mobility spectrum will have been generated, but the user does not need to interpret the spectrum for alarm notification.

6.1.6 System Status

The system must indicate when the instrument is, or is not, ready to operate within normal measurement parameters.

6.1.7 Radiation Control

If the detector contains a radioactive source for ionization, the source is sealed and there is no radiation exposure hazard, as determined by qualified personnel.

6.1.8 Power

The system can operate from a standard AC power outlet and has a power on/off switch.

6.1.9 Environmental

The system must be operable under the normal conditions of indoor environments, and within the temperature range 0°C to 40° C and between 1 % and 90 % relative humidity (non-condensing).

² Minimum amounts defined by detector challenge levels reported in ROA 837-36-04.

6.1.10 Set-up From Receipt

The manufacturer provides clear instructions for the user to set up the instrument and bring it to a state of operational readiness, and may provide training (free or at cost) on or off-site for such a purpose.

6.1.11 Instrument Use and Maintenance

There is an instrument manual or equivalent document that describes the proper use and maintenance of the instrument, the average time involved in such maintenance, and any consumable items required for use and maintenance. The manufacturer provides support to resolve any problems with the use and maintenance of the instrument.

6.2 Additional Performance Criteria

6.2.1 Additional compounds detected

The explosives AN, NG, HMX, DNT, Tetryl, TATP, and UN are among the common explosives that can be detected by IMS in addition to the required HE compounds.

Multimode Applications:

6.2.2 Compounds detected

Narcotics that can be detected may include: Cocaine, Heroin, Amphetamine, Methylenedioxy Amphetamine (MDA), Methamphetamine (MDMA), Methylenedioxy Ethylamphetamine (MDEA), THC, Morphine, Econidine Methylester, Tetrahydrocannabinol (THC), Phencyclidine (PCP) and LSD.

Chemical warfare (nerve) agents that can be detected may include: Tabun, Sarin, Soman, Cylcosarin, Mustard gas, Lewisite, and Agent VX.

6.2.3 Mode switch

The system is either capable of switching from one mode to another, or can simultaneously analyze in positive and negative ion mode.

Improved Accuracy

6.2.4 Selectivity

The system must correctly and unambiguously identify the 3 required high explosives; and there can be no *erroneous identifications*. There may be a membrane added for selectivity, or an additional analysis technique, such as Gas Chromatography (GC) or Mass Spectrometry (MS) may be attached to the instrument to improve accuracy of compound identification.

6.2.5 Data recording/output

There is an easily accessible recording of the alarm, either in digital form or on printed medium, containing a date/time stamp, the explosive(s) detected, and the peak intensity. There is access to the spectrum that generated the alarm.

Mobile Applications

6.2.6 Portability

The system must be able to withstand the physical rigors of transportation and the routine shocks and vibrations incurred by bumping, etc. during operation. It must be of a dimension and weight that allow for portability.

6.2.7 Power

For mobile applications, the system should also operate from battery power. The system is able to operate continuously from battery power for a minimum of 60 minutes. A 12V DC cigarette lighter adaptor is useful for mobile applications employing a vehicle.

6.2.8 Environmental

The system must be fully encased for protection against water. The system must be able to respond to rapid and/or considerable changes in barometric pressure, and must be able to operate for extended periods of time under high humidity conditions. The system must be operable in outdoor environments that include moderate to high levels of dust and other airborne particles.

Screening Applications

6.2.9 False positive alarm rate

The false positive alarm rate must not be greater than 5%.

6.2.10 Startup Time

The system startup time is 1 hr or less.

6.2.11 Throughput Rate

The throughput rate is 180 samples/hour when no alarms are present.

6.2.12 Data recording/output

There is an easily accessible recording of the alarm, either in digital form or on printed medium, containing, at a minimum, a date/time stamp and the explosive(s) detected.

6.2.13 Continuous operation time

The system is available to analyze samples 95% of the time during a 24-hour period.

6.2.14 Repeatability

In a series of rapidly repeated measurements on standard test materials, the false negative alarm rate should approach 0%.

6.2.15 Reproducibility

In a protracted series of measurements on standard test materials taken across several days and in a variety of environmental conditions, the false negative alarm rate should approach 0%.

7. TEST METHOD

The test method utilizes a kit containing solutions of the three required HEs (RDX, PETN, and TNT), and is designed to test the basic performance criteria of IMS trace explosives detectors. The test method kit was developed by the Transportation Security Laboratory of the Transportation Security Administration at the William J. Hughes Technical Center and will be available commercially shortly (contact information will go here when available).

Safety note: The HEs, when in solution, do not constitute an explosives hazard. The amount of solid HE deposited on the test swipe after evaporation of the solvent is on the order of nanograms, which is not considered an explosives hazard. The solvent used in the test solutions is isobutyl alcohol, and care should be taken to avoid unnecessary exposure of personnel to the solvent or the HEs. Material Data Safety Sheets (MSDSs) for the solvent and the three HEs are included in this document. Please use the MSDS sheets and other relevant documents to become familiar with health and safety issues connected with use of this IMS test method.

Contents of Test Method Kit (Figure 9):

- 1) Three plastic squeeze bottles containing solutions of one HE + isobutyl alcohol, and one plastic squeeze bottle of the solution blank (isobutyl alcohol). Each bottle holds 6 mL.
RDX concentration: $0.14\ \mu\text{g/mL}$, or $\approx 3\ \text{ng per drop}$
TNT concentration: $1.0\ \mu\text{g/mL}$, or $\approx 23\ \text{ng per drop}$
PETN concentration: $1.7\ \mu\text{g/mL}$, or $\approx 40\ \text{ng per drop}$
- 2) Supports for sample swipes (4 plastic holders with circular cutouts).
- 3) Test method (this document).
- 4) MSDSs for isobutyl alcohol, RDX, TNT, and PETN.

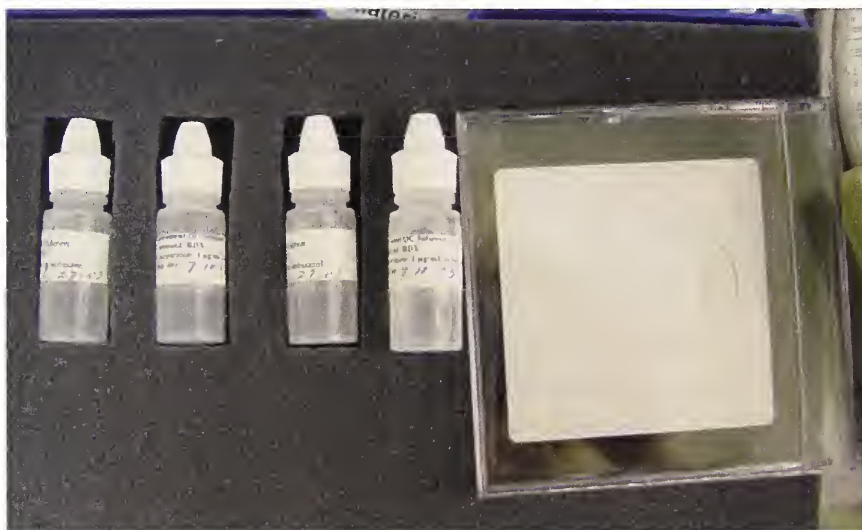


Figure 9. Test method kit.

Storage Requirements and Solution Details:

The HE solution concentrations are established by NIST Report of Analysis 837-36-04. The average volume of a drop from a squeeze bottle is 23 μ L, dispensed by holding the bottle at approximately a 45° angle. The HE test solutions can be stored at room temperature and have a shelf life exceeding 4 months. The solutions should be discarded in a safe manner when less than 1 mL remains.

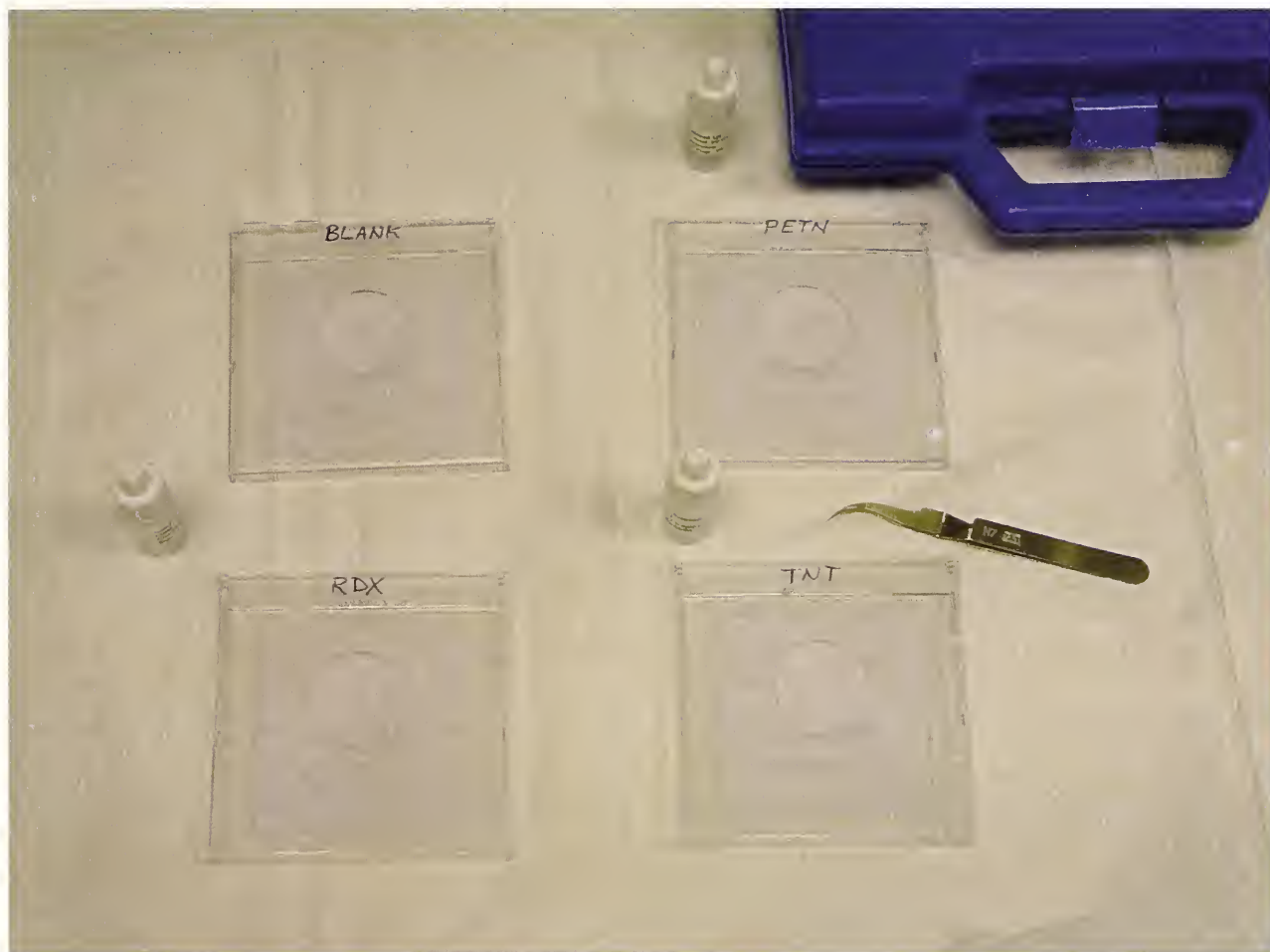


Figure 10. Workspace preparation for use of IMS test method kit.

Procedure:

Workspace preparation (Figure10):

- 1) Wear gloves.
- 2) Cover table of bench surface with clean, absorbent, disposable material.
- 3) Provide convenient and sanitary area for disposal of used swipes and other consumables.
- 4) Label each plastic support for one of the 4 solutions to reserve each for a single HE, or the blank, to prevent cross contamination.

- 5) Have suitable containers ready to hold prepared test samples.

Preparation of test samples (Figure 11):

- 1) Place a sample swipe over the circular cutout on the sample swipe support. It is important that the portion of the swipe that accepts the test solution is not in contact with any surface that might serve to wick away the solution. A different type of sample support may be used as long as the general requirement of supporting the swipe above a surface is met.
- 2) Choose one of the explosive test solutions. Before using the squeeze bottle, ensure the cap is closed tightly and shake well.
- 3) Open the cap, hold the bottle at about a 45° angle from horizontal, and squeeze the bottle slowly to deposit *one drop* on the specified sample swipe. The solution must be deposited within the target area for sample deposition as indicated by the manufacturer (Figure 12). *If more than one drop is deposited, discard the sample swipe and begin again.* (The analyst may wish to practice this step beforehand.)
- 4) Wait until the solution is visibly dry on the sample swipe. Drying times vary from approximately 5 minutes to 20 minutes or longer, depending on the specific sampling swipe used and the ambient temperature and relative humidity. Avoid using temperatures above 30 °C to accelerate the drying process.
- 5) Once the sample wipe is dry it may be analyzed immediately or stored individually in a clean, closed, labeled container (Figure 13) and held at room temperature for up to 4 hours. While RDX is relatively stable at room temperature, IMS responses for PETN and TNT will significantly degrade over time.
- 6) Repeat with the remaining 3 test solutions to prepare 3 additional test samples. If the test sample prepared with the sample blank is to be stored prior to analysis, ensure that the container in which it is stored is clean and has not previously been used for positive test samples.

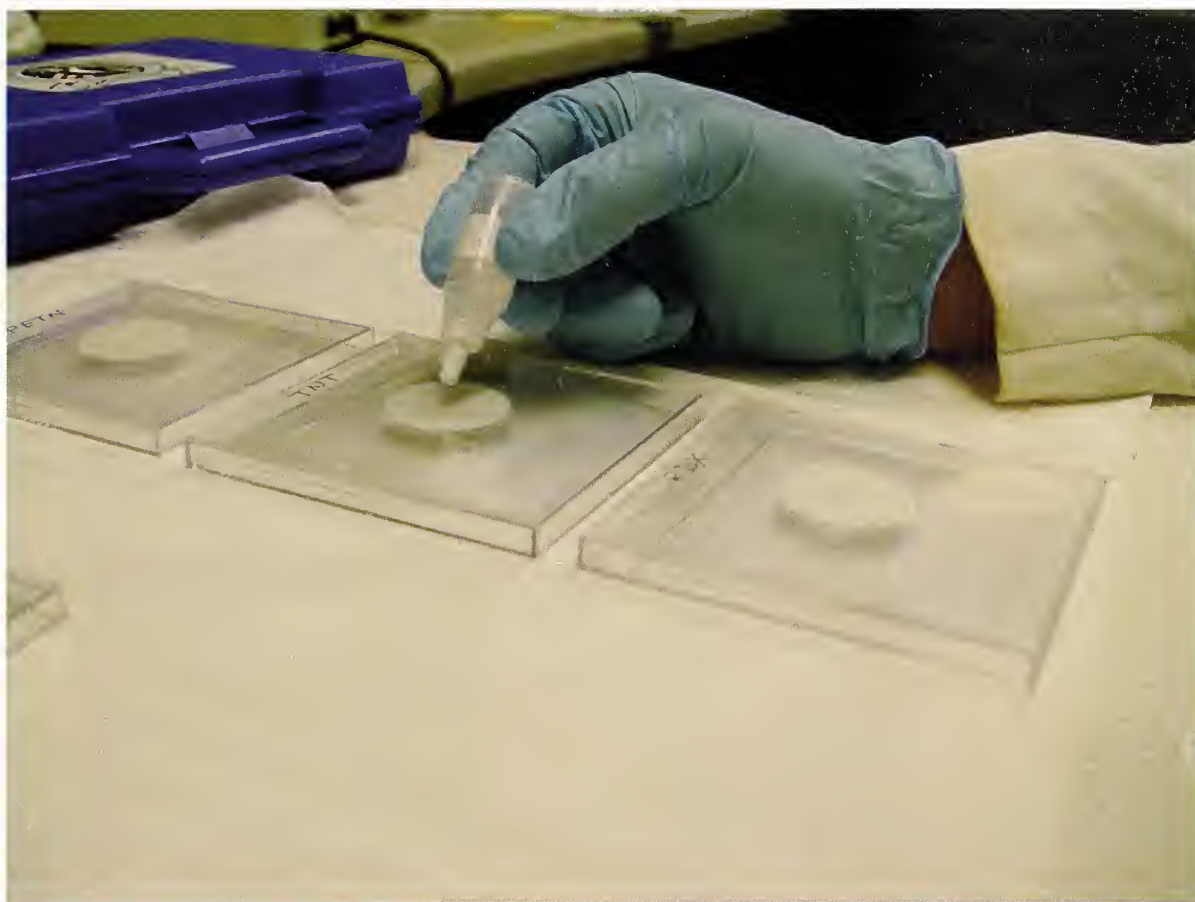


Figure 11. Test sample preparation. Application of single drop holding the bottle at approximately a 45° angle.

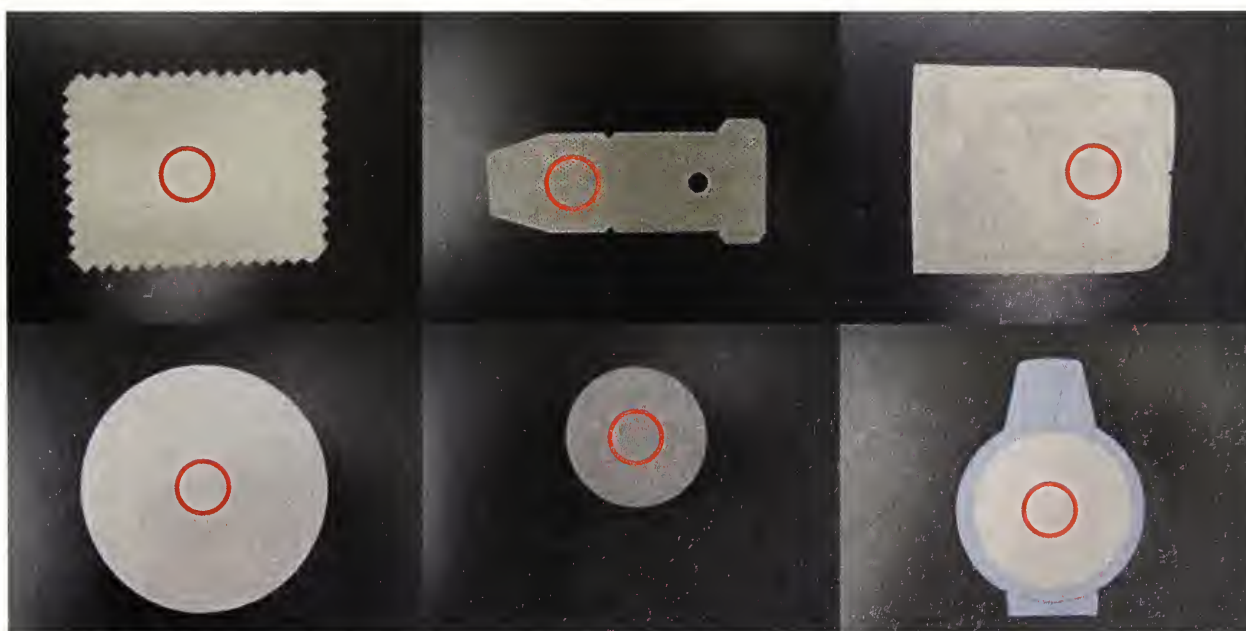


Figure 12. Some examples of available sample swipes for different IMS trace explosives instruments. Red circles mark the target area for sample deposition.

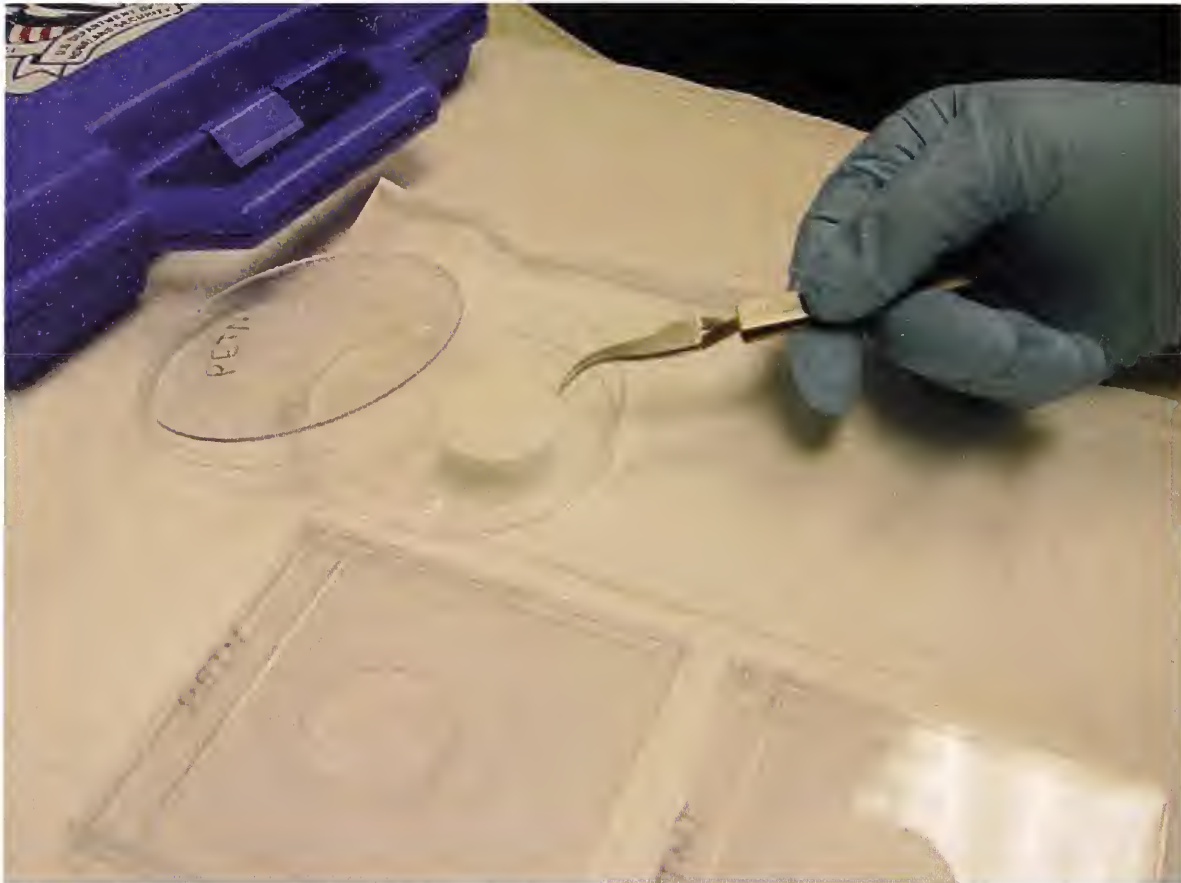
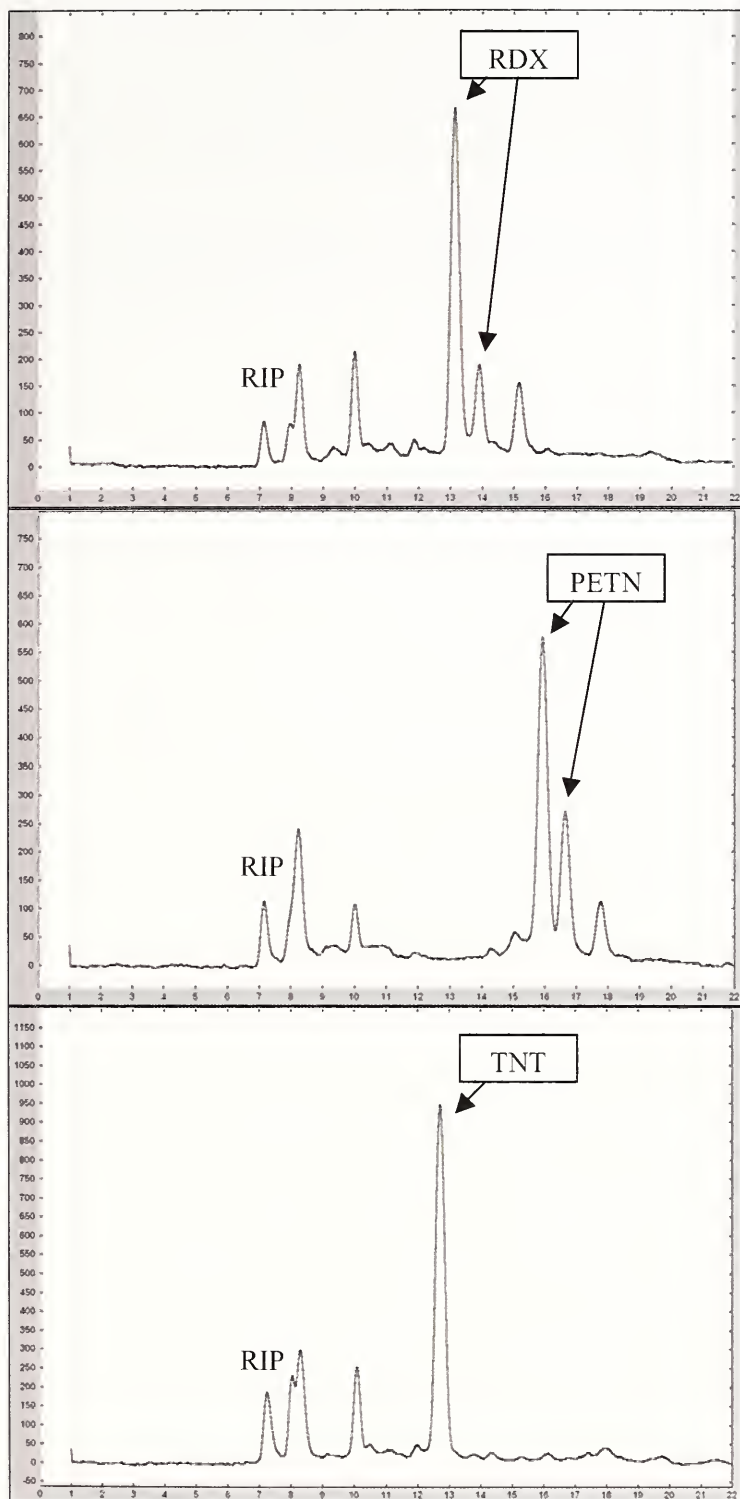


Figure 13. Storage of dried test sample.

Analysis of test samples:

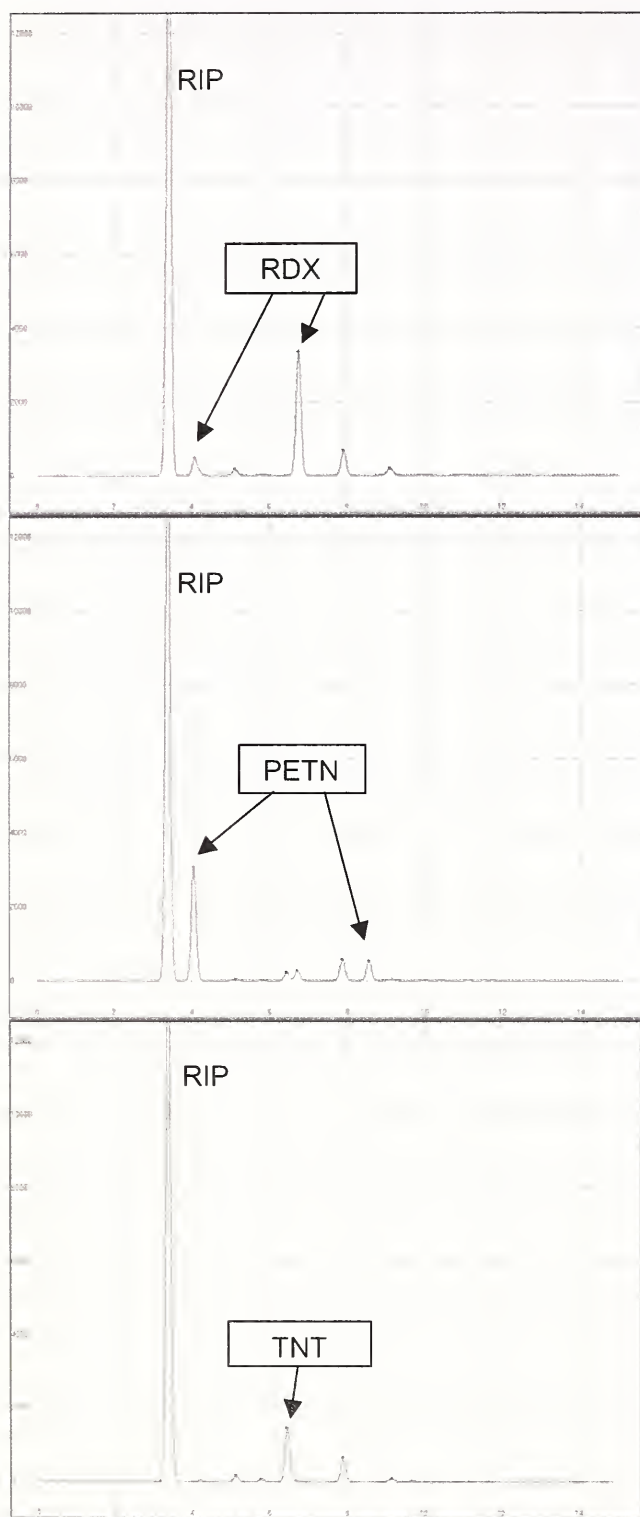
- 1) Ensure that the IMS trace detector is at *operational readiness*.
- 2) Analyze the blank and confirm a negative test.
- 3) Analyze one of the HE test samples and confirm a positive alarm and the identity of the explosive tested.
- 4) Repeat steps 1 through 3 with remaining 2 positive test samples.
- 5) Repeat the measurements of the 3 HE test samples several times to verify that the false negative response rate for this test is 0%.

The ion mobility spectra of the HE test samples may look like those shown in Figure 14 if the IMS system does *not* contain a membrane, or like those in Figure 15 if the IMS system does contain a membrane.



Raw single plasmagrams from an open (non-membraned) instrument. Here, the reactive ion peak (RIP) is relatively small, leading to the formation of multiple cluster ions for RDX and PETN. Note the unlabeled peaks between the RIP and the explosive peaks. These unidentified peaks are associated with the sampling media and are common in open systems.

Figure 14. Examples of IMS spectra plotted as intensity vs. drift time in milliseconds obtained from test method samples on an instrument without a membrane. Labeled peaks include the reactive ion peaks (RIP) and the explosives peaks.



Processed plasmagrams from a closed (membraned) instrument. The peaks for RDX and PETN correspond to the parent peak (on right at longer drift times) and a nitrate decomposition peak (on left near RIP). In this instrument, the desorption and inlet temperatures are set high (220 °C) to optimize the RDX parent peak, which causes decomposition and loss of sensitivity for PETN.

Figure 15. Examples of IMS spectra plotted as intensity vs. drift time in milliseconds obtained from test method samples on an instrument with a membrane. Labeled peaks include the reactive ion peaks (RIP) and the explosives peaks.

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GE Vaportracer²

User's Manual, Document number MA001074, Rev. 2, 2003.

APPENDIX 1. Definitions Specific To IMS Trace Detection

Analyte : A specific substance that is undergoing analysis or is being measured.

Auto-calibration : A *calibration* procedure that occurs without operator input. Typically the first event that follows warmup of the unit. Utilizes an internal *calibrant*.

Calibrant : A material used to perform a *calibration*.

Calibration : The process of comparing the response of an instrument against a standard to determine instrument accuracy or to make a correction. For IMS instruments, calibration is the establishment of accurate drift times and peak intensities for known material(s).

Detection limit : See *Limit of Detection*.

Desktop : A general category of IMS trace detectors that is meant to be positioned on a horizontal surface during operation. Samples are obtained by swiping and are brought to the instrument for analysis. The primary mode of operation is particle analysis. Some desktop models are more portable than others, and could be used in vehicles, etc.

Desiccant : A substance that has a high affinity for water and is used as a drying agent for the air in the IMS.

Desorber : A device that removes vaporizable molecules for introduction into a gas stream, usually by heating. IMS detectors that use particle sampling, such as desktop and portal systems, have a desorber.

Dopant : A reactive substance added to the sample that improves the sensitivity and selectivity of the IMS instrument for explosive compounds. A common dopant is dichloromethane.

Erroneous identification : A true *positive alarm*, but where the explosive is misidentified. For example, the IMS instrument reports TNT instead of RDX.

False positive alarm : A *positive alarm* for a sample that does not contain any explosives.

False negative : A failure to detect the presence of a sufficient quantity of explosive.

First Responders : Personnel who are members of organizations and agencies that respond to all manner of emergency events, and who respond to, manage, and work to prevent terrorist events involving weapons of mass destruction. They include emergency communications centers; emergency medical services, fire, and rescue; hazmat teams; law enforcement agencies; bomb squads; SWAT; hospitals; public health; risk management; security; emergency management and disaster management; transportation and public works; gas, water and electric companies; the American Red Cross; and members of the U.S. Coast Guard and U.S. Customs and Border Protection.

Hand-Held : A general category of IMS trace detectors that is meant to be carried during operation. Samples are obtained by drawing air into the detector, but there may also be accommodation for particle-based analysis using sample swipes. The primary mode of operation is direct vapor analysis.

High Explosives (HE) : Explosives characterized by a very high rate of reaction, high pressure development, the presence of a detonation wave in the explosive, and which can be caused to detonate by means of a blasting cap when unconfined.

Ion Mobility Spectrum : Graphical data produced by Ion Mobility Spectrometry (IMS). The data result from charge transfer of the ionized gas molecules to the detector and are plotted as intensity with respect to drift time. Spectrum peaks are identified on the basis of drift time and concentration is proportional to peak intensity.

Limit of Detection : The limit of detection (LOD) is commonly accepted as the smallest amount of a substance that can be reliably detected by a measurement process. The analytical signal from this amount must be high enough above background variations to give statistical confidence that the signal is real, so the LOD is normally defined as the amount of substance that generates an average signal three times higher than the standard deviation of the background.

Low Explosives : Explosives that are characterized by deflagration (a rapid combustion that moves through an explosive material at a velocity less than the speed of sound), not detonation.

Manual Calibration : A calibration that is user initiated. Usually performed to test the internal calibration, or for general quality control. Utilizes an external *calibrant*.

Marking Agent: A volatile compound added during manufacture of explosives (usually at very low concentration) to enhance detection of explosives by vapor detection. The 4 marking agents included in the International Civil Aviation Organization (ICAO) Convention on the Marking of Plastic Explosives for the Purpose of Detection are EGDN, DMNB, p-MNT and o-MNT (defined in Appendix 2). See *Taggant*.

Measurand : A quantity or property subjected to a measurement process. The test sample prepared by the method given in this document is an example of a measurand.

Mode Switch : Setting the IMS instrument to a new detection mode, as in switching from explosives detection to narcotics detection, or from particle sampling to vapor sampling.

Nuisance alarm : A positive alarm that occurs due to the detection of an explosive when the presence of the explosive is due to either sample contamination or other nonthreatening or innocuous circumstances.

Operational cycle : The process that occurs after a successful startup and that includes sampling, analysis, and return to readiness.

Operational readiness : The status of the IMS detector after a successful startup. The IMS detector is ready to accept samples for analysis.

Plasmagram: See *Ion Mobility Spectrum*.

Portal : A device designed as a walk-through unit that analyzes explosives or narcotics by IMS. Samples are collected as people pass through the portal either by using air jets to dislodge particles or by using natural convection.

Positive Alarm : A signal to warn of the presence of an explosive. The alarm can be visual and/or auditory. The alarm is triggered in the IMS when a peak is detected above the preset threshold level.

Reactive Ion Peak (RIP): The relatively intense peak, commonly having a short drift time, which is comprised mainly of chloride ion clusters through ionization of the dopant.

Reliability : A device may be termed reliable when its response(s) to samples are accurate and reproducible throughout its useful life.

Repeatability : Closeness of the agreement between the results of successive measurements of the same *measurand* carried out subject to all of the following conditions: the same measurement procedure; the same observer; the same measuring instrument, used under the same conditions; the same location; repetition over a short period of time.

Reproducibility : Closeness of the agreement between the results of measurements of the same *measurand*, where the measurements are carried out under changed conditions such as: principle or method of measurement; observer; measuring instrument; location; conditions of use; time.

Return to Readiness : The process that follows the analysis of a sample that insures the detector is ready to accept the next sample. Following the analysis of a positive sample, this process includes a test to confirm a return to background levels, and may require cleaning, bakeout, or other remedial actions.

Sample swipes : Pads that are made from various types of materials, including fabric and paper, which are used to collect particle samples for desktop and some handheld units. In addition to a variety of materials, there are a variety of shapes and sizes of swipes for the different instrument models.

Startup Time : The time involved in bringing the instrument from a cold start to operational readiness.

Selectivity : The capability of a method or instrument to identify a target substance or sample constituent in the presence of nontarget substances. [e.g. the ability to identify explosives when other materials are present.]

Sensitivity : The capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. [The ability to detect small amounts of a compound.]

Taggant A solid, liquid, or vapor-emitting substance put into an explosive material for the purposes of detection or identification. See marking agent.

Threshold level : A response level set for a given explosive above which a *positive alarm* is activated. The threshold level is significantly higher than the detection limit for that explosive.

Throughput rate : The number of distinct samples that can be processed in a given period of time.

Traceability : The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

Trace Explosive : Very small amounts of explosives left on surfaces after handling explosives, e.g. in fingerprint residues, or present as vapors in the air. As opposed to bulk explosives, e.g. a bomb or other visually obvious amounts.

Vapor Concentration : The actual abundance of a gaseous compound in air, which is dependent upon several factors including the *vapor pressure* and the physical state (solid, liquid, or gas) of the compound, temperature, and diluting effects of ambient air and wind. The vapor concentration can be several orders of magnitude below the *vapor pressure*, and might not be detectable by current IMS technology.

Vapor Pressure : The pressure of a vapor in a closed container when in equilibrium with its liquid or solid form at a specified temperature. It is a measure of the tendency of a material to form a vapor. The higher the vapor pressure, the higher the potential vapor concentration.

APPENDIX 2. Glossary of Selected Explosives Materials

AN - Ammonium nitrate (NH_4NO_3) Used as a solid oxidizer in explosive mixtures such as ammonium nitrate-fuel oil (ANFO), which was used in the Oklahoma City bombing.

Black powder - The original form of gunpowder containing an alkali nitrate (usually potassium or sodium), charcoal, and sulfur. Currently, black powder is very difficult to analyze by IMS due to the inability to vaporize potassium or sodium nitrate at typical desorber temperatures.

C-4 – A US military plastic-bonded explosive containing RDX as the HE.

Detasheet® – A flexible explosive manufactured in sheet form, and containing PETN as the HE.

DMNB – 2,3-Dimethyl-2,3 dinitrobutane ($\text{C}_6\text{H}_{12}\text{N}_2\text{O}_4$) One of the 4 marking agents identified in the International Civil Aviation Organization (ICAO) Convention on the Marking of Plastic Explosives for the Purpose of Detection.

DNT - Dinitrotoluene ($\text{C}_7\text{H}_6\text{N}_2\text{O}_4$) Commonly used as a flash inhibitor in double- or triple-base smokeless powders.

EGDN - Ethylene glycol dinitrate ($\text{C}_2\text{H}_4\text{N}_2\text{O}_6$) An HE that has been used in mixtures with NG. One of the 4 marking agents identified in the International Civil Aviation Organization (ICAO) Convention on the Marking of Plastic Explosives for the Purpose of Detection.

Gunpowder – See black and smokeless powder.

HMX - Cyclotetramethylene tetranitramine ($\text{C}_4\text{H}_8\text{N}_8\text{O}_8$) Also known as octogen. An HE that is commonly associated with RDX.

MNT - Mononitrotoluene ($\text{C}_7\text{H}_7\text{NO}_2$) Either para (p-MNT) or ortho (o-MNT). Marking agents identified in the International Civil Aviation Organization (ICAO) Convention on the Marking of Plastic Explosives for the Purpose of Detection.

NG - Nitroglycerine ($\text{C}_3\text{H}_5\text{N}_3\text{O}_9$) An HE used in formulations such as dynamite and multi-base smokeless gunpowder.

PETN - Pentaerythritol tetranitrate ($\text{C}_5\text{H}_8\text{N}_4\text{O}_{12}$) An HE used in formulations such as Semtex.

RDX - Cyclotrimethylene trinitramine ($\text{C}_3\text{H}_6\text{N}_6\text{O}_6$) Also known as cyclonite or hexogen. An HE used in formulations such as Semtex and C-4.

Semtex formulations – A plastic-bonded explosive of variable composition typically containing RDX and PETN.

Smokeless powder – The modern form of gunpowder. The bulk component of the propellant is nitrocellulose; double-base and triple-base powders may also contain NG (nitroglycerine),

nitroguanidine, and DNT (dinitrotoluene). Smokeless powders contain primers, plasticizers and stabilizers in addition to the propellant. Double- and triple-base powders analyzed by IMS may alarm on NG and/or DNT.

TATP - Triacetone triperoxide ($C_9H_{18}O_6$) An unstable, highly sensitive explosive manufactured from common chemicals such as acetone, peroxide, and acid. Used in a mixture with PETN by attempted shoe bomber Richard Reid.

Tetryl - Trinitrophenylmethylnitramine ($C_7H_5N_5O_8$) An HE used as a detonator. Manufactured and used primarily from WWI through WWII.

TNT - Trinitrotoluene ($C_7H_5N_3O_6$) An explosive used commonly for military purposes.

UN – Urea nitrate $CO(NH_2)_2NO_3$ An explosive made from urea and nitric acid; used in the Bali, Indonesia bombing.

